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(21) International Application Number: PCT/US97/04404 (22) International Filing Date: 19 March 1997 (19.03.97) (30) Priority Data: 08/618,881 20 March 1996 (20.03.96) US (71) Applicant: APPLIED SPECTROMETRY ASSOCIATES, INC. [US/US]; W226 N555G Eastmound Drive, Waukesha, WI 53186 (US). (72) Inventors: KAHLE, Scott, J.; 4468 North Sawyer Road, Oconomowoc, WI 53066-3330 (US). BEEMSTER, Bernard, J.; 10062 North Sunnycrest Drive, Mequon, WI 53092-5419 (US). REYNOLDS, Bruce, A., M.; N54 W26326 Lisbon Road, Sussex, WI 53089 (US). WEST, Kevin, J.; 1868 Haymarket Road, Waukesha, WI 53186 (US). (74) Agent: EHRMANN, Thomas, W.; Quarles & Brady, 411 East Wisconsin Avenue, Milwaukee, WI 53202 (US).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: METHOD FOR ANALYZING AMMONIA IN WATER		
(57) Abstract <p>A method is disclosed for conditioning a water or wastewater sample for analysis of ammonia using a UV spectrometer. The sample is conditioned by adding a solution of sodium hydroxide to raise the pH to above 10. Ethylene diamine tetraacetic acid may be added prior to or with the sodium hydroxide solution to prevent hardness precipitation. Thereafter, a solution of sodium hypochlorite is added to the sample in an amount sufficient to react with all of the ammonia present to form chloramines. In using the conditioned sample, an unconditioned sample is first introduced into the UV spectrometer and an absorbance pattern at multiple wavelengths is measured. The conditioned sample is then introduced to the UV spectrometer and an absorbance pattern at the same multiple wavelengths is measured. The absorbance pattern of the unconditioned sample is subtracted from that of the conditioned sample to produce an absorbance pattern indicative of the amount of ammonia present in the sample.</p>		

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METHOD FOR ANALYZING AMMONIA IN WATER

Background of the Invention

This invention relates to the analysis of the amount of ammonia present in water and wastewater, and particularly to a method of on-line analysis for ammonia using ultraviolet (UV) spectrometers.

Ammonia analysis in water and wastewater has generally been performed by using ion-specific electrodes, or by using single wavelength colorimetry following the addition of phenol or mercury iodide. These reagents are highly toxic and require special handling and sample disposal.

A method for analyzing water and wastewater that does not require the use of reagents employs a UV spectrometer. Although pure water is transparent to light in the ultraviolet and visible wavelength ranges (from 200 to 700nm) and much of the very near infrared wavelength range (from 700 to 1400nm, with two exceptions), the presence of certain chemical constituents in the water will result in absorbance of light within specific wavelength ranges. Each chemical constituent has its own unique absorbance pattern, or signature. If multiple chemical constituents are present, the individual light absorbance patterns will combine in a manner that produces an absorbance pattern that is a product of all absorbing constituents in the water. Furthermore, the intensities of the absorbance pattern is proportional to the concentration of the chemical constituents that produce the pattern. With a light source of measurable intensity and a water sample with known path length, pattern recognition analysis can be performed using statistical and mathematical analysis techniques to identify the presence and concentration of specific chemical constituents in the water.

UV spectrometers used for such analysis typically include: one or more light sources that are capable of emitting a portion of the ultraviolet wavelength spectrum; a means for conducting the light from the source to the sample which may employ fiber optic cables, lenses, slits, prisms, mirrors, or other optical components; a sample chamber which permits entry and exit of a sample stream and transmission of light through a portion of the sample, or reflection of light by the sample, or diffraction of light within the sample; a means for collecting and conducting light from the sample to a monochromator which may employ fiber optic cables, lenses, slits, prisms, mirrors, or other optical components; a monochromator to disperse the spectrum of light into component wavelengths and to focus the light for detection; a photon detector or detectors capable of rapid transduction of numerous individual wavelengths across a wavelength range of interest, which may or may not be an array detector, to convert light intensity into an electrical signal; a processor to convert the electrical signals from the detector into digital data; and a microprocessor to perform mathematical operations, including the calculation of the concentration of chemical substances in the water sample, to store and execute operational instructions for the analyzer system, to store calibration files, and to supervise digital communication functions.

Certain chemical substances, such as nitrates or nitrites, will naturally absorb ultraviolet light in unique patterns over a range of wavelengths. However, other chemical substances, such as ammonia, do not exhibit natural light absorbance characteristics, or have natural light absorbance characteristics too weak to reliably detect, or have absorbance patterns that are affected by factors such as pH. Water and wastewater containing ammonia requires sample conditioning before detection by a UV spectrometer.

Summary of the Invention

A method is disclosed for conditioning a sample of water or wastewater for analysis of ammonia by a UV spectrometer which includes the steps of raising the pH of the sample to above 10 and preferably to 11 or 12, and mixing a hypochlorite solution with the sample to react with the ammonia to form chloramines. The hypochlorite solution is added in an excess amount relative to the ammonia concentration to ensure complete reaction of the ammonia. The treated sample is ready for analysis by a UV spectrometer.

Preferably, the pH of the sample is raised by mixing a solution of sodium hydroxide (NaOH) with the sample. A hardness complexing agent such as a solution of a sodium salt of ethylene diamine tetraacetic acid (EDTA) may be added with the sodium hydroxide solution or before to prevent hardness precipitation in the water sample. The hypochlorite solution preferably uses sodium hypochlorite, common household bleach.

Also in accordance with the invention, the sample conditioning method may be incorporated into a method for analysis of water and wastewater that includes analyzing a sample of the water or wastewater in a UV spectrometer prior to conditioning to determine the absorbance of the sample. This establishes a base line for absorbance of chemicals and impurities other than ammonia. A second sample of water that is conditioned as described above is then introduced into the UV spectrometer which measures and stores the absorbance pattern of the conditioned sample. The absorbance pattern of the sample before conditioning is subtracted from the absorbance pattern of the conditioned sample. The resultant signature pattern is a combination of the hypochlorite and chloramine absorbance, independent of

background absorbance. This absorbance signature pattern is used to calculate the ammonia concentration, preferably using advanced pattern recognition techniques.

Preferably, the light absorbance is detected over multiple wavelengths to provide improved absorbance signatures.

5 An object of the invention is to provide a method for the analysis of ammonia in water and wastewater that does not require the use of undesirable toxic reagents.

Another object of the invention is to provide a method for the testing of water and wastewater for ammonia that uses conditioned samples fed to UV spectrometers.

10 A further object of the invention is to provide a method for conditioning samples of water and wastewater for UV spectrometry analysis for ammonia that uses commonly available chemicals.

The foregoing and other objects and advantages of the invention will appear in the following detailed description of the invention. In the description, reference is made to the accompanying drawings which illustrate a preferred embodiment of the invention, including preferred apparatus and graphical representations of the results of the testing.

Brief Description of the Drawings

Fig. 1 is a schematic functional diagram of a sample conditioning unit and UV process analyzer useful for carrying out the method of the invention; and

20 Fig. 2 is a graph of typical absorbance pattern of ammonia resulting from the methods of the present invention.

Detailed Description of the Preferred Embodiment

The method of the present invention is particularly suited for use with a UV spectrometer model UV-6100 Process Analyzer manufactured by Applied Spectrometry Associates, Inc. of Waukesha, Wisconsin. The process analyzer is a multiple

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wavelength ultraviolet absorbance spectrometer designed to function continuously as an on-line instrument. The analyzer is capable of detecting any chemical substance that absorbs light in the ultraviolet (and blue visible) wavelength range. A total of 256 individual wavelengths are simultaneously detected by projecting light through a sample as it passes through a flow cell. The absorbance at these 256 wavelengths define an absorbance pattern of a solution, and the pattern is a function of the chemical composition of the solution. Pattern recognition (sometimes called "chemometrics") is used to extract information concerning the presence and concentration of specific chemicals in a solution from the detected absorbance signature for the solution. Using multiple wavelengths provides improved results. There is no dependence on a narrow wavelength band which might be influenced by absorbing substances other than those being sought.

Referring to Fig. 1, a light source 10 in the form of a xenon flash lamp is used to generate light at all wavelengths across the ultraviolet wavelength range and into the visible wavelength range. A fiber optic cable 11 conveys the light to a flow cell 12 where the light is transmitted through a sample of water or wastewater. A fraction of the light is absorbed by the chemicals in the sample, and the remaining light is conveyed through a second fiber optic cable 13 to a spectrograph assembly 14 which includes a 256 element photodiode array detector that segments the detection range into 256 equal intervals. An instrument control board 15 includes a microprocessor. The board 15 receives the output of the photodiode array detector and controls the UV light source and the spectrograph assembly 14. A backlit LCD display 16 and a keypad 17 of push buttons are connected to the control board 15 to permit adjustments of the microprocessor. An RS-232 serial port 18 is available for digital

communications. Data logging is accomplished using a dedicated 4-20 mA communication link 19.

Because ammonia does not naturally absorb light in the ultraviolet wavelength range, chemical conditioning is required for analysis using ultraviolet spectrometry.

5 Furthermore, ammonia in water exists in equilibrium between ammonia (NH_3) and ammonium (NH_4^+). The ammonia predominates at a pH near 10 and above. The present method for analysis of ammonia in water avoids the need to perform distillations prior to analysis and also avoids the use of toxic reagents such as phenol or mercury iodide. The method substitutes the use of an oxidizer (hypochlorite) and
10 an alkaline solution to provide a known pH range in the sample. The oxidizer and alkaline solutions are introduced into the sample in a conditioning unit, as shown in Fig. 1.

The conditioning unit includes a mixing chamber 25 to which a sample of wastewater can be directed through an inlet valve 26. A portion of the sample can be
15 sent through a bypass line 27 to an outlet valve 28 for connection directly to the flow cell 12. Injector pumps 29 and 30 provide injected volumes of the alkaline solution and the oxidizer solution, respectively, to the mixing chamber 25. A peristaltic pump 31 pumps the mixed sample from the mixing chamber 25 through the outlet valve 28 to the flow cell 12.

20 In carrying out the methods of this invention, two solutions are prepared in advance: a 1.25 molar NaOH/EDTA solution, and a .75-.80% NaOCl solution. The NaOH and EDTA solution is prepared by mixing 500 grams of NaOH pellets and 250 grams of EDTA with 10 liters of deionized water. The NaOCl solution is prepared by mixing 1.5 liters of a 5.0 to 5.25% NaOCl solution in 8.5 liters of deionized water.
25 A preferred source for the NaOCl solution is common household bleach and

particularly Clorox brand blue label bleach. The solutions may also be made using sample water, distilled water, or tap water.

A sample of water is pumped from a sample stream and flushed through the inlet valve 26, the mixing chamber 25, the peristaltic pump 31, and the outlet valve 28 into the flow cell 12. The sample flow must be adequate to fully flush the tubing and the flow cell 12 to prevent carry-over from previous samples. The UV light source is activated, and the absorbance of the water sample in the flow cell 12 is measured by the spectrograph assembly 14 and stored for later use. The mixing chamber 25 of the sample conditioning unit is filled with a second sample of water from the sample stream. Once the mixing chamber 25 is full, a small quantity of the NaOH/EDTA solution is injected into the mixing chamber 25 by the injector pump 29 (eg. 5ml of the solution to 500 ml of water sample). The EDTA binds up the hardness to eliminate precipitation that could result as the NaOH raises the pH of the sample. The pH of the sample is raised above 10 and preferably above 11, but in any event in the range of 10 to 12. The EDTA may be injected separately into the sample before the NaOH solution, but a solution of both chemicals is effective and eliminates an additional step.

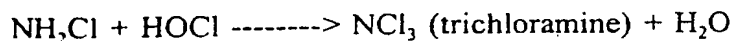
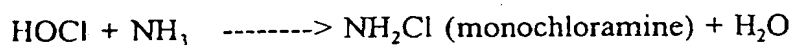
A small quantity of the NaOCl solution is then injected by the pump 30 into the mixing chamber 25 to react with the ammonia in the sample to form chloramines (eg. 5 ml of the solution to 500ml of water sample). The NaOCl is added in excess amounts relative to the ammonia concentration to ensure complete reaction of the ammonia. The solution is allowed to react for two minutes and the conditioned sample is then pumped by the peristaltic pump 31 to the flow cell 12.

The UV light source 10 is again activated, and the spectrograph assembly 14 measures the absorbance signature of the reacted water sample which is also stored.

The absorbance signature of the sample before conditioning is subtracted from the absorbance signature of the conditioned sample. The resultant signature is a combination of the hypochlorite and chloramine absorbance, independent of background absorbance. The absorbance signature is used to calculate the ammonia concentration, preferably using advanced pattern recognition techniques. The ammonia concentration is displayed to absolute terms on the display 16.

Fig. 2 shows a series of typical absorbance patterns of conditioned samples having various concentrations of ammonia. The absorbance patterns of Fig. 3 are plots of wavelength in nanometers versus absorbance. The first dip in the plot, at approximately 240nm represents the ammonia concentration. The high absorbance "bump" centered at approximately 290nm is the result of the bleach.

The reaction of chlorine and ammonia in dilute aqueous solutions forms three types of chloramines:



These reactions are in general by steps, so that they compete with each other. A series of complex reactions with all of these substances involves the chlorine substitution of each of the hydrogen atoms in the ammonia molecule. These reactions are dependent on pH, temperature, contact time, and initial chlorine to ammonia ratio. Which form of chloramine is present is not a certainty. However, by controlling the pH, volumes and contact time, the signatures from one sample to another will be consistent and the amount of ammonia can be determined.

As thus far described, the purpose of introducing an unconditioned sample of water or wastewater into the UV spectrometer was to set a base absorbance pattern.

However, the absorbance of the unconditioned sample can also be used to determine the concentrations of chemical constituents that have a natural absorbance of ultraviolet light without the need for conditioning. For example, the unconditioned sample could be used to determine the concentrations of nitrate or total oxidized nitrogen. After the absorbance pattern of the unconditioned sample is subtracted from that of the conditioned sample to determine the concentration of ammonia, it can be added back to produce an absorbance pattern for total dissolved nitrogen.

The method of the invention provides a reliable and accurate analysis of ammonia in water and wastewater. The methods rely upon commonly available chemicals to condition water samples and eliminate the need for costly and toxic reagents.

We claim:

1. A method for conditioning a sample of water or wastewater for detection of ammonia by a UV spectrometer comprising the steps of:

mixing a quantity of a solution of sodium hydroxide with the sample in an amount sufficient to raise the pH of the sample to above 10; and

5 mixing a quantity of a solution of sodium hypochlorite with the sample in an amount sufficient to react with all of the ammonia present to form chloramines.

2. The method of claim 1 together with the step of mixing an ethylene diamine tetraacetic acid solution prior to or together with the sodium hydroxide solution to prevent precipitation in the sample.

3. The method of claim 1 wherein the sodium hydroxide solution is added in an amount sufficient to raise the pH of the sample to 11 or 12.

4. A method for conditioning a sample of water or wastewater for detection of ammonia by a UV spectrometer, comprising:

raising the pH of the sample to above 10; and

5 mixing a quantity of a hypochlorite solution with the sample in an amount sufficient to react with all of the ammonia present to form chloramines.

5. The method of claim 4 wherein the hypochlorite is sodium hypochlorite.

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6. The method of claim 4 together with the step of mixing a solution of a hardness complexing agent prior to or together with the sodium hydroxide solution to prevent precipitation in the sample.

7. The method of claim 6 wherein the hardness complexing agent is the disodium salt of ethylene diamine tetraacetic acid.

8. A method for conditioning a sample of water or wastewater for detection of ammonia by a UV spectrometer comprising the steps of:

mixing a quantity of a solution of sodium hydroxide and the disodium salt of ethylene diamine tetraacetic acid with the sample in an amount sufficient to raise the pH of the sample to above 10; and

mixing a quantity of a solution of sodium hypochlorite with the sample in an amount sufficient to react with all of the ammonia present to form chloramines.

9. A method for conditioning a sample of water or wastewater for detection of ammonia by a UV spectrometer, comprising:

raising the pH of the sample to 11 or 12; and

mixing a quantity of a solution of sodium hypochlorite with the sample in an amount sufficient to react with all of the ammonia present to form chloramines.

10. A method of analyzing water or wastewater for detection of the amount of ammonia present in the water comprising the steps of:

passing a first sample of the water through a UV spectrometer;

measuring the absorbance of the first sample;

5 conditioning a second sample of the water by raising the pH of the second sample above 10 and thereafter injecting a quantity of a hypochlorite solution into the water sample in an amount sufficient to have the ammonia react with the hypochlorite to form chloramines;

passing the conditioned second sample through the UV spectrometer;

10 measuring the absorbance of the conditioned second sample; and

subtracting the absorbance of the first sample from the absorbance of the conditioned water sample.

11. The method of claim 10 wherein the pH is raised by the addition of a sodium hydroxide solution.

12. The method of claim 10 wherein the pH of the conditioned sample is raised to 11 or 12.

13. The method of claim 11 wherein a small quantity of an ethylene diamine tetraacetic acid solution is added to the second sample prior to or along with the addition of the sodium hydroxide solution.

14. The method of claim 10 wherein the hypochlorite is sodium hypochlorite.

15. The method of claim 13 wherein the measurement of the absorbance of the first sample is used to determine the concentration of chemical substances having a natural absorbance of ultraviolet light.

16. A method of analyzing water or wastewater for detection of the amount of ammonia present in the water comprising the steps of:

passing a first sample of the water through a UV spectrometer;

measuring the absorbance pattern of the first sample at multiple wavelengths
5 of light;

conditioning a second sample of the water by raising the pH of the second sample above 10 and thereafter injecting a quantity of a hypochlorite solution into the water sample in an amount sufficient to have the ammonia react with the hypochlorite to form chloramines;

10 passing the conditioned second sample through the UV spectrometer;
measuring the absorbance pattern of the conditioned second sample at the same multiple wavelengths; and

subtracting the absorbance pattern of the first sample from the absorbance pattern of the conditioned water sample.

17. The method of claim 16 wherein the hypochlorite solution is formed from sodium hypochlorite

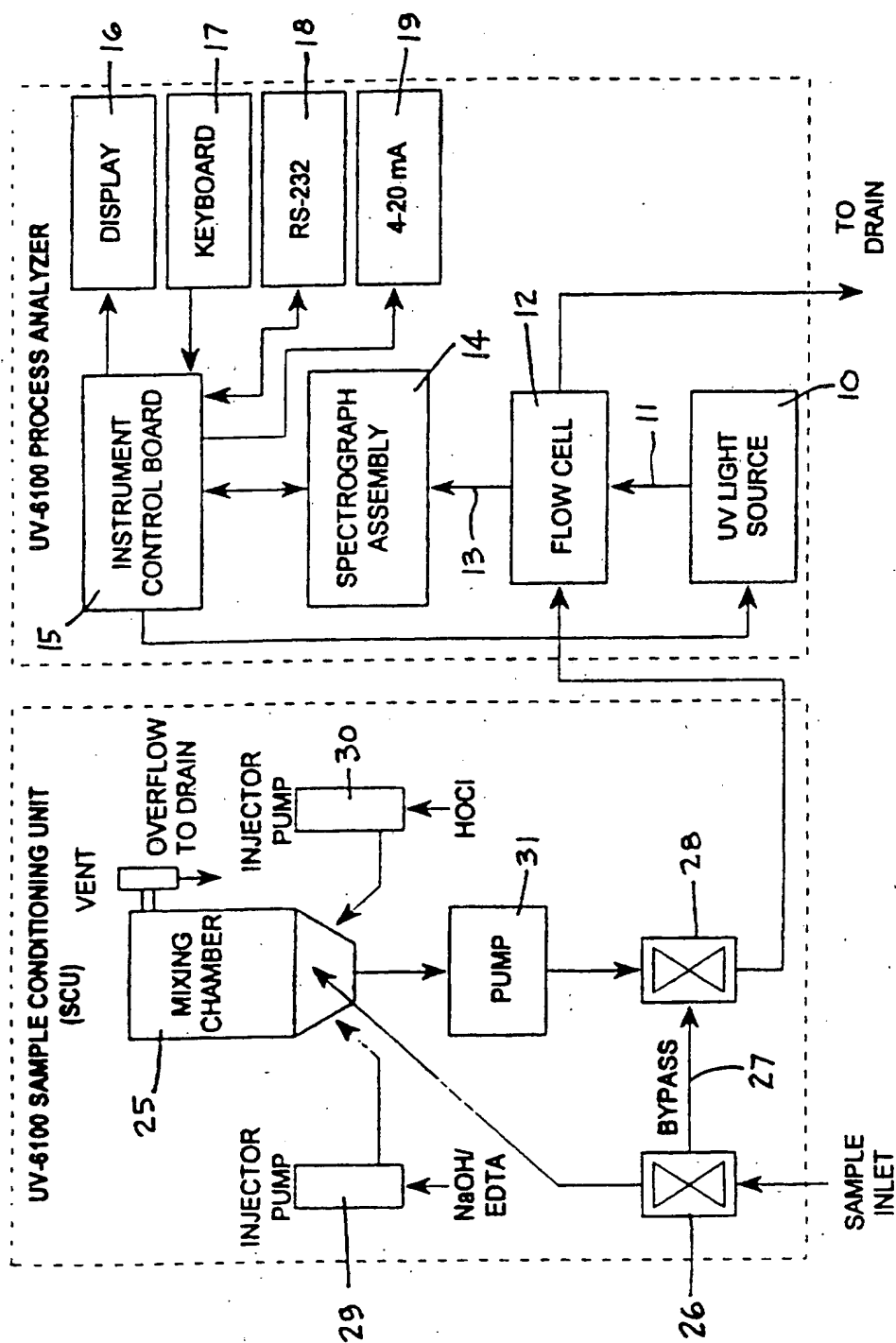


FIG. 1

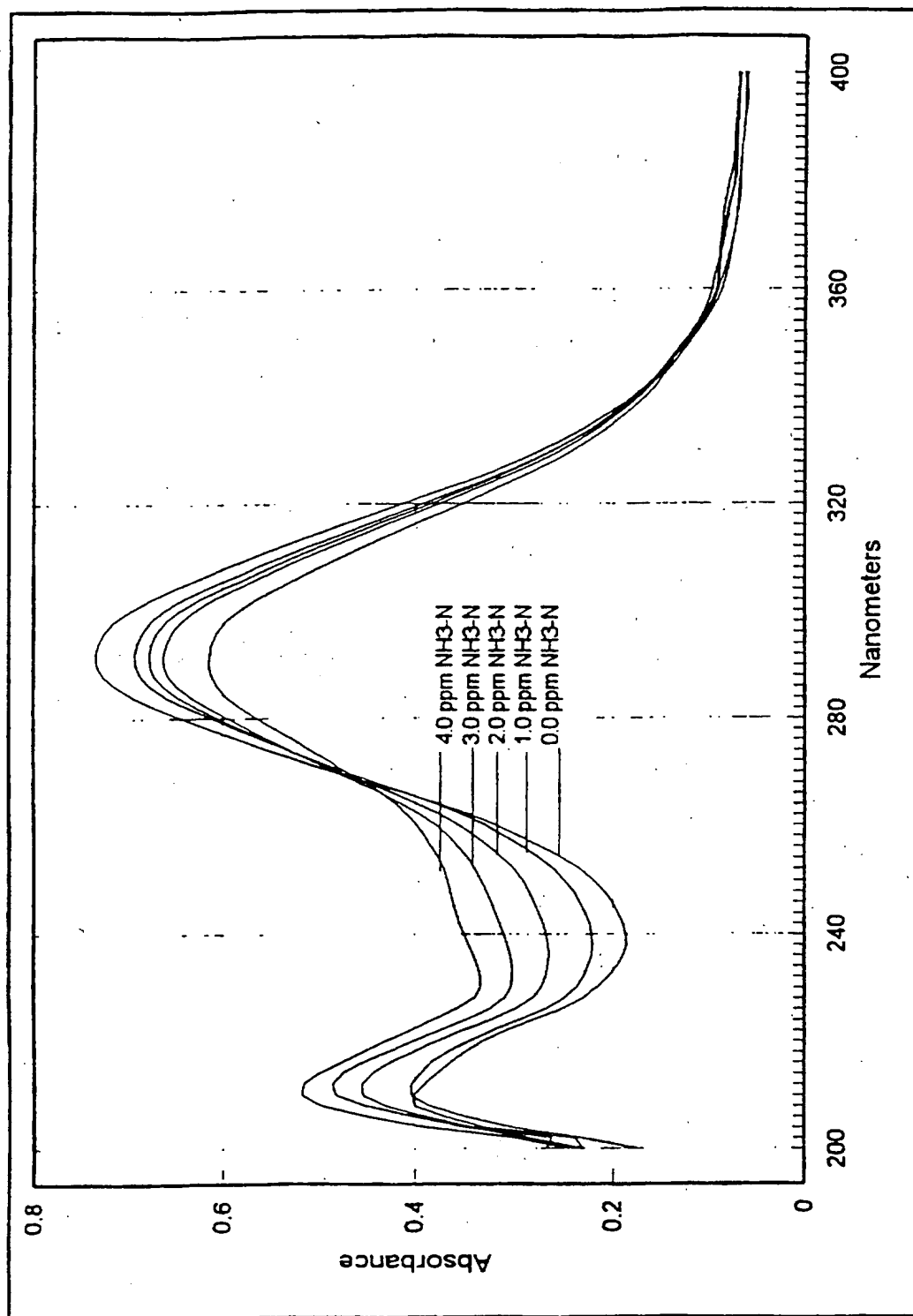


FIG. 2

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US97/04404

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : G01N 33/18

US CL : 436/106, 111, 113, 124, 125, 164, 166

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 436/106, 111, 113, 124, 125, 164, 166

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Please See Extra Sheet.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 94/02836 A (BRIGGS) 03 February 1994, see entire document.	1-17
Y	S. J. Kahle et al "HAES- a Hybride Absorption/Emission Spectrometer for On-line Multicomponent Analysis of Liquids" Advances in Instrument Control, 1992, Vol. 47, pages 1287-1294, see entire document.	1-17
Y	M. Anbar et al, "The Hydrolysis of Chloramine in Alkaline Solution" Journal of the American Chemical Society, May 1962, Vol. 84, No. 10, pages 1790-1796, see entire document.	1-17



Further documents are listed in the continuation of Box C.



See patent family annex.

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	F. W. Czech et al, "Determination of Mono-, Di-, and Trichloramine by Ultraviolet Absorption Spectrophotometry" Analytical Chemistry, May 1961, Vol. 33, No. 6, pages 705-707.	1-17
A	C. Colin et al, "Les Chloramines en Solution: Preparations, Equilibres, Analyse" Analisis 1987, Vol. 15, No. 6, pages 266-274.	1-17

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US97/04404

B. FIELDS SEARCHED

Electronic data bases consulted (Name of data base and where practicable terms used):

STN search terms: sodium hypochlorite, potassium hypochlorite, hypochlorite, naocl, kocl, ocl, ammoni####, nh3, nh4, chloramine#, det##, detect?, determin?, testing, measur?, monitor?, scns?, analysis, uv, ultraviolet, ultra violet, online, on line, monochloramine, dichloramine, trichloramine